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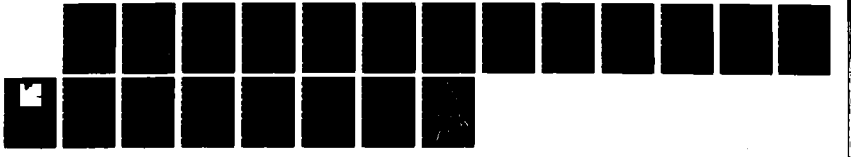

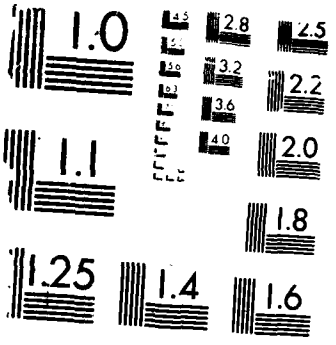

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CHEMICAL DEGRADATION AND STRESS CRACKING OF POLYCARBONATE IN DS2

LIDIA H. LEE and JANICE J. VANSELOW

POLYMER RESEARCH DIVISION

September 1987

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ABSTRACT

Polycarbonate (PC) undergoes loss of physical integrity and ultimate dissolution in the decontaminant DS2. In order to gain a better understanding of this process, this study evaluates the effects of each of the three components of DS2 on PC. Weight loss measurements of PC in DETA (70%), MCEL (28%) and MCEL+NaOH (2%) show that DETA is the most aggressive component in DS2. The FTIR spectra of PC/DETA, PC/MCEL, and PC/MCEL+NaOH indicate that the principle degradation mechanism of PC in DS2 is the chain cleavage reaction taking place at the carbonate linkage due to the attack of the amine and the hydroxide. Mass spectroscopy analysis on the dry PC/DS2 residue indicates that numerous low molecular weight products are formed. Results of stress cracking of PC with DS2 and DETA show a considerable breakdown in resistance. The MCEL component is less aggressive than DETA, but still induces significant stress cracking.

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INTRODUCTION

DS2 is sometimes considered a universal decontaminant in the sense that it will rapidly hydrolyze both vessicants and nerve agents, although it may be ineffective against biological warfare agents. The usual decontamination procedure involves washing the contaminated object with DS2 for two hours. Such an operation could cause serious corrosive effects on some materials due to the nature of DS2, which consists of 70% diethylene triamine (DETA), 28% methyl cellosolve (MCEL) and 2% NaOH by weight (0.5 molar). The problem is particularly prominent in applications where thermoplastic materials are used. For instance, polycarbonate, used widely for aircraft windows, helicopter canopies and transparent armor because of its good impact strength, optical transparency and high toughness, is extremely susceptible to the attack of DS2. It undergoes dissolution readily in DS2, resulting in complete loss of physical integrity if not properly coated with a chemical resistant coating.[1] Mylar, a thermoplastic polyester, also shows a large reduction of weight upon exposure to DS2.[2] A detailed study of the interaction between DS2 and peroxide cured neoprene has been reported.[3] The report demonstrates the multicomponent aspects of the transport behavior of DS2 in the action of decontaminating a crosslinked polymer. The results of the study, however, may not be generally applicable to glassy thermoplastic polymers, because interactions other than thermodynamic swelling, as in a crosslinked elastomer, may take place. The purpose of this work is to study the interactions between DS2 and PC by examining the effects of the individual components of DS2 and their combinations on the properties of polycarbonate, and to advance the present understanding of the interaction mechanisms between the decontaminant and polymers.

EXPERIMENTAL

Sorption uptake was determined at room temperature as a function of time on samples ~1 inch square and 0.25 inch thick. Samples were immersed in a DS2 solution contained in a screw cap jar, one sample to a jar. At preselected intervals, the samples were removed, blot dried on filter paper, and transferred to a tared weighing bottle.

A stress cracking study was done by placing samples (6" x 1" x 0.25") on a three-point-bending fixture. Testing and data handling follow the recommendations of ASTM D 790. For all tests, the support span is 16 times the depth of the specimens. The maximum strain occurs at midspan, and may be calculated as follows:

$$r = 6\,Dd/L^2$$

where:

r = maximum strain in the outer layers (mm/mm),
D = maximum deflection of the center of the specimen (mm),
L = support span (mm), and
d = depth (mm).

1. LEE, I.H. *Chemical Resistance of Parylene C Coated Transparent Thermoplastics*. CRDC Report, September 1985.
2. *NBC Material Handbook*. Roland Technical Report No. ROL-0109-L, p. 3-89 - 3-92.
3. SCHNEIDER, N.S., ROSEN, B., and MEDDON, I. *The Role of Transport Variables in the Action of DS2 on an Elastomer*. CRDC Report, 1984.

The time it took for the first craze to appear after the application of the chemical was taken as the craze initiation time.

DISCUSSION

Figure 1 shows the immersion results (weight loss vs square-root of time) of PC in DETA, MCEL, and MCEL+NaOH. It can be seen that DETA is the most active component in DS2, causing the most rapid rate of PC weight loss. While MCEL alone caused a very slight weight loss of PC, the addition of NaOH increased its reactivity significantly, as evidenced by the fact that the sample lost about 48% of its original weight within 48 hours in MCEL+NaOH. The fact that the data plotted in this form are initially linear and then appear to level off suggests the presence of a diffusion controlled mechanism in the eroding process. As the eroding process of PC in MCEL+NaOH continued with time, the solution became more dense and opaque. Therefore, the levelling off was most likely due to reaching the saturation point of PC in the solution. These results indicate that NaOH plays an active role in the dissolution process of PC in DS2, and that MCEL, being relatively inert to PC, might function as a carrier for NaOH in the polymer medium, as previously suggested.[3] Figure 2 shows the weight loss of PC in DS2 and in DS2 without NaOH (DS2*). It can be seen that the rate of weight loss in DS2* is nearly the same as that in DS2. The result is somewhat surprising, since the combined action of DETA and NaOH in DS2 is expected to cause the dissolution process to accelerate. The results suggest that while NaOH alone is capable of causing PC to undergo dissolution, it does not affect the overall eroding strength of DS2 on PC.

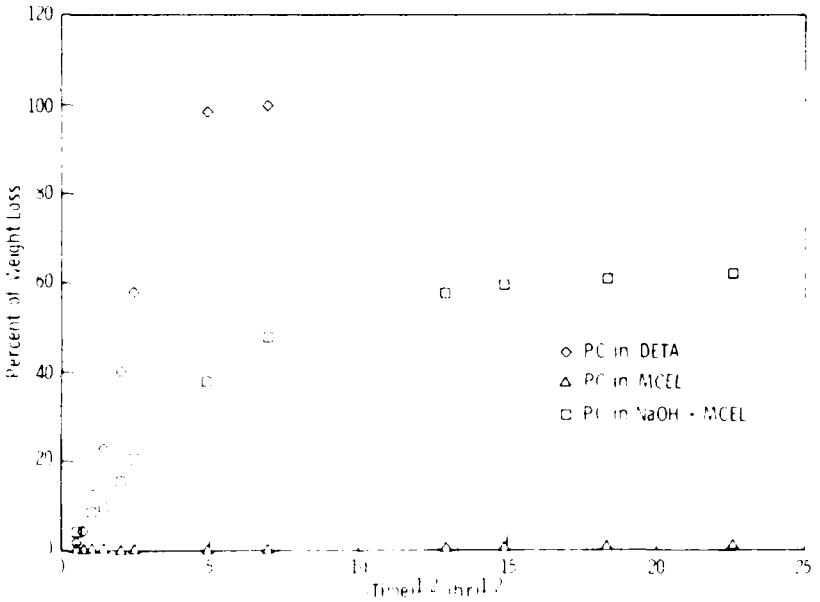


Figure 1. Immersion test results of PC in DETA, MCEL and MCEL+NaOH.

The weight loss of PC in DS2 could be due to physical dissolution as a result of the similar solubility parameters of PC [23.0 $(J/cm^3)^{1/2}$] and DS2 [estimated around 22.9 $(J/cm^3)^{1/2}$], or certain kinds of chemical hydrolysis and nucleophilic reactions that cause the weight loss of PC by a cleavage reaction. To clarify the exact mechanism involved, Fourier Transform Infrared Spectroscopy (FTIR) was used to

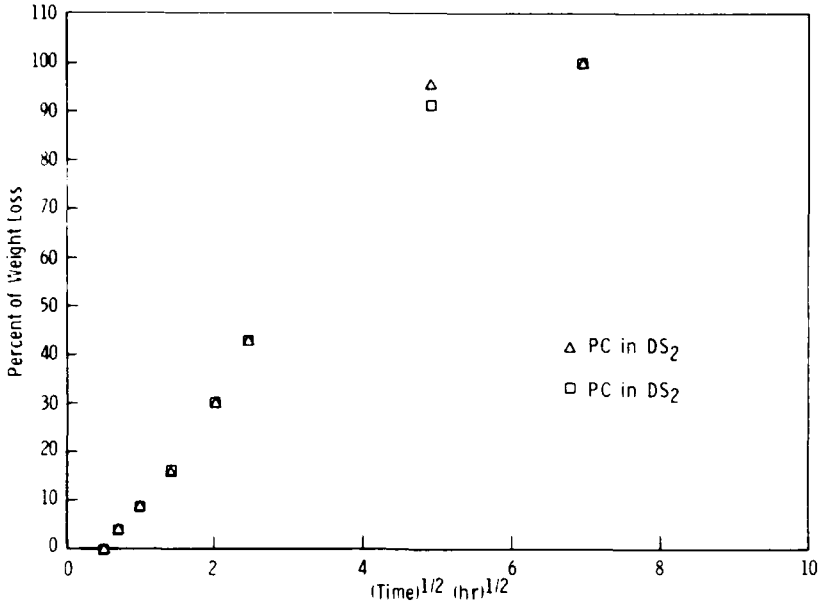
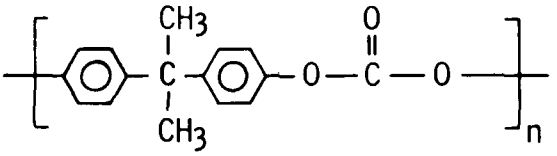


Figure 2. Immersion test results of PC in DS2 and DS2*.

monitor any chemical structural changes of PC in DS2 and its components. The most probable site for any chemical reaction to take place in PC, with the structure



is at the carbonate group (-OCOO-). This group typically gives the absorption band at 1780 cm^{-1} on the IR spectrum. Figure 3 illustrates the IR spectrum of neat PC. The presence of this absorption band can be used to verify the occurrence of chemical degradation of PC in DS2. Furthermore, any reaction product that might form as a result of the interaction between PC and the components of DS2 could be identified by analyzing the absorption bands. Experiments were done by withdrawing liquids from the jars in which PC was immersed in the components of DS2 or their combinations. In most cases, liquids were drawn after PC had lost more than 50% of weight in the jar. The exception was with PC immersed in MCEL. The weight loss of PC in this liquid is very minute, and the liquid was drawn after about two weeks of immersion. The liquids were then injected into a cell made with two pieces of KBr salt plates or smeared onto the salt plate for FTIR analyses.

Interactions With DETA

Figure 3 shows the IR spectrum of PC/DETA, with the spectra of pure DETA and neat PC as references. It is evident that the absorption band at 1780 cm^{-1} (corresponding to carbonate groups) could not be detected, but a new absorption band at 1700 cm^{-1} (corresponding to a different sort of carbonyl compound) emerged, suggesting the formation of a new chemical group in the system. There was concern that the absence of the carbonate peak was due to the concentration of PC in DETA being too low. Mass spectroscopy, however, confirmed that long molecular chains of PC had

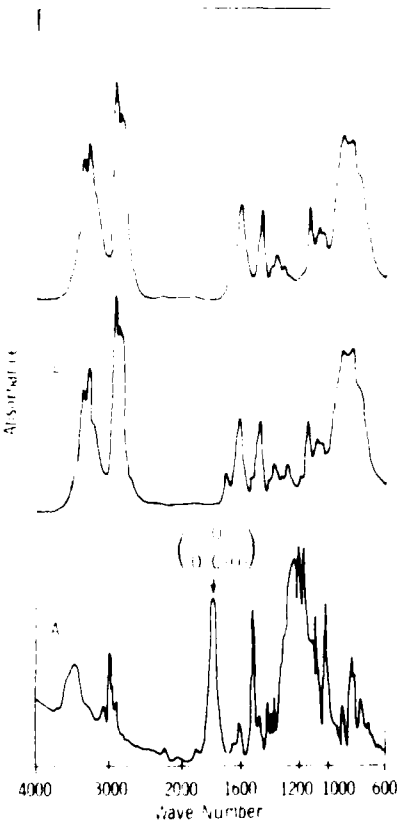


Figure 3. FTIR spectrum of the interactions of DETA with neat PC. A) neat PC, B) PC/DETA solution, and C) pure DETA.

broken down into low molecular weight products, as will be discussed later. It can be concluded, based on these results, that the erosion of PC in DETA was due to the nucleophilic attack of DETA on the carbonate groups of PC, most likely by the primary amine, resulting in the chain cleavage of PC.

Interactions With MCEL and NaOH

The solution of PC/MCEL has an IR spectrum nearly identical to that of pure MCEL as shown in Figure 4. The result is not surprising, since PC showed little weight loss in MCEL. As mentioned previously, the addition of NaOH in MCEL caused PC to lose more than 50% of its original weight within a few days. Figure 4 shows the IR spectrum of PC/MCEL+NaOH solution as well as the IR spectrum of MCEL. The region around the carbonate peak (1780 cm^{-1}) for the PC/MCEL+NaOH solution also appeared flat, indicating that most, if not all, of the carbonate groups had undergone some kind of chemical reactions, as seen in the case of PC/DETA. Several new peaks resulted corresponding to the new chemical groups formed. The results of the IR analyses confirm the fact that NaOH participates actively in the chain cleavage reaction of PC in DS2.

Interactions With DS2

Figure 5 shows the spectrum of PC/DS2, with the spectra of DS2 and PC as references. The DS2 spectrum seems to be a composite of the DETA and MCEL spectra,

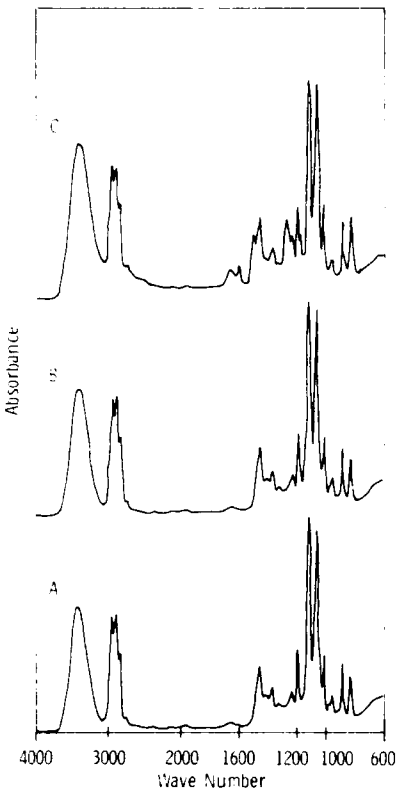
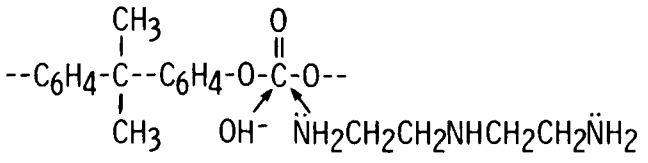


Figure 4. FTIR spectrum of the interactions of MCEL and NaOH with PC. A) pure MCEL, B) PC/MCEL solution, and C) PC/MCEL+NaOH solution.

suggesting that there is little chemical interaction between DETA and MCEL. The PC/DS2 spectrum, however, looks very similar to the spectrum of PC/DETA, shown previously in Figure 3. This could be due either to the large quantity of DETA present in the DS2 or the fact that DETA is the most aggressive component in DS2 in the chain cleavage process. To eliminate any possible obscuring effects caused by the presence of the solvents, the IR spectra of the dry reaction products were obtained by driving off the DS2 on a hot plate. Results are shown in Figure 6, with 6b representing the degraded PC. The information obtained was similar to that for the case where solvent was present; i.e., the carbonate groups had disappeared as a result of interaction with DS2, confirming, as previously suggested, that erosion of PC in DS2 is mainly due to the chain cleavage reaction rather than simple thermodynamic dissolution.

The results of the mass spectroscopy analysis on the dry PC residue obtained by driving off DS2 indicated that numerous products with molecular weights lower than 300 were formed as a result of the following interactions:



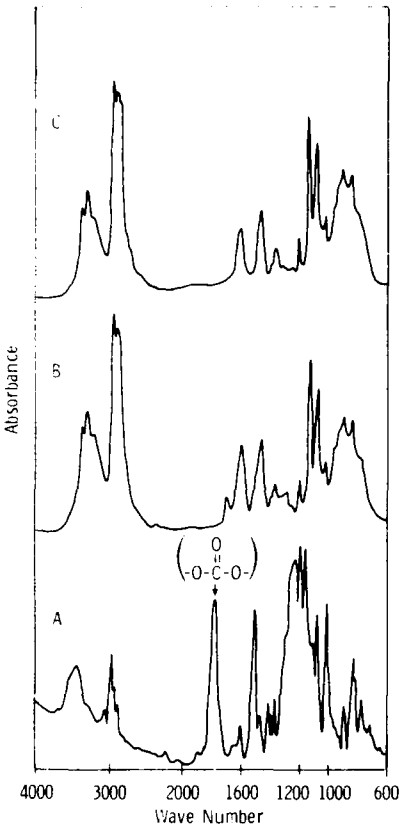


Figure 5. FTIR spectrum of the interactions of DS2 with PC. A) neat PC, B) PC/DS2 solution, and C) pure DS2.

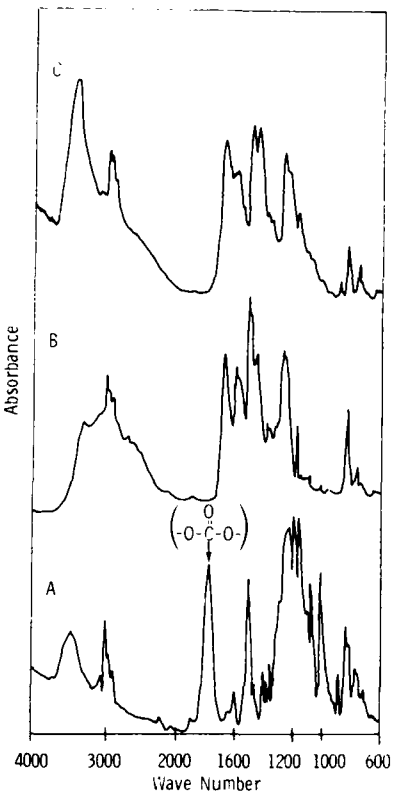


Figure 6. FTIR spectrum of dried DS2 and PC/DS2 solution. A) neat PC, B) dried PC/DS2 solution, and C) dried DS2 solution.

The large number of compounds formed from these reactions makes the interpretation of the results obtained by mass spectroscopy rather complicated and difficult. However, it was possible to identify some of the products formed; examples are carboxylic acid derivatives and phenols.

Stress Cracking

While it may take a period of a few hours or days to see the corrosive effect of DS2 on PC, crazes or cracks can form in PC in a few seconds if stress is simultaneously applied to PC. The stress could either be mechanical or simply residual stresses due to material processing. It is generally accepted that absorption of the liquid and concomitant Tg reduction are intimately related to stress cracking behavior. Several authors[4-6] have shown correlation between the critical crazing strain of a polymer and the equilibrium solubility of the liquid in the polymer, thereby predicting environmental stress cracking of the polymer from solubility considerations. The concept has been extended to include a hydrogen bonding parameter

4. BERNIER, G. A., and KAMBOUR, R.P. *The Role of Organic Agents in the Stress Crazing and Cracking of Poly(2,6-dimethyl-1,4-phenylene oxide)*. Macromolecules, v. 1, 1968, p. 393.
5. KAMBOUR, R.P., GRUNER, C.L., and ROMAGOSA, E.E. *Bisphenol-A Polycarbonate Immersed in Organic Media: Swelling and Response to Stress*. Macromolecules, v. 7, no. 2, 1974, p. 248.
6. IMAI, Y., and BROWN, N. *Environmental Crazing and Intrinsic Tensile Deformation in Polymethylmethacrylate*. J. Mater. Sci., v. 11, 1976, p. 417.

to aid in predicting stress cracking characteristics of several different polymers.[7] Organic agents with solubility parameters similar to those of a polymer can act either as solvents or cracking agents. When the difference is somewhat larger, the organic liquids act as crazing agents. Although the theories for predicting the stress cracking behavior of a polymer in an organic liquid have been established, there has been little study done for the cases in which chemical chain cleavage is involved.

The study of the stress cracking of PC in contact with DS2 was done by bending the uncoated PC specimens in a three-point-bending fixture at various strain levels. The time it took for the first craze to appear after the application of the chemicals was taken as the craze initiation time. The results indicate that the craze initiation time decreases with the applied strain. A threshold strain, termed critical strain, was noted for each chemical tested, below which no craze or crack was observed for a relatively long period of time (~1 hour), as shown in Figure 7. A higher critical strain means a better resistance of the material to the chemical tested. The critical strain of dry PC in the air[4] (i.e., in the absence of any kind of unfavorable environment) is about 1.9%, below which no crazes could form in a short period of time. Table 1 summarizes the critical strain values of PC in DS2 and its components.

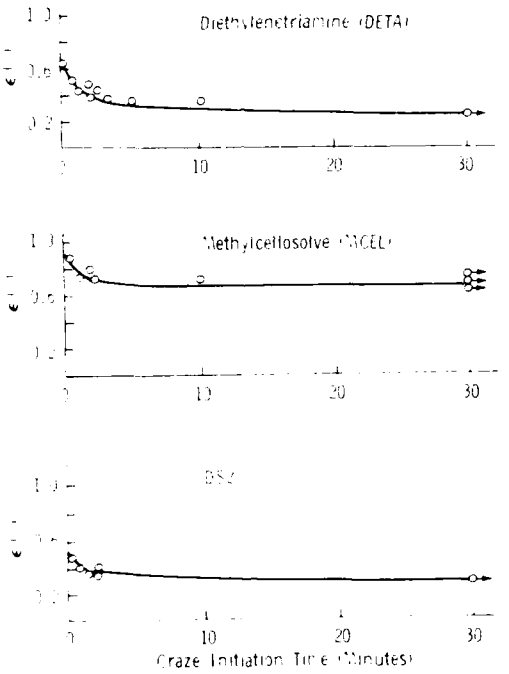


Figure 7. Craze initiation time vs. applied strain.

It is apparent that the presence of the liquids lowers the resistance of PC to stress cracking significantly. Upon contact with DS2, cracks developed rapidly in PC and propagated to the edges, as shown in Figure 8. The process of chain cleavage generates weak spots in the PC specimen that act as stress concentrators, promoting

[7] VINCENT, P.I. & ERAHA, S. *Influence of Hydrogen Bonding on Crazing and Cracking of Amorphous Thermoplastics.* Polymer, [illegible]

Table 1. STRESS CRACKING RESISTANCE OF PC

Chemical	Critical Strain (%)
DS2	0.32
DETA	0.34
MCEL	0.68

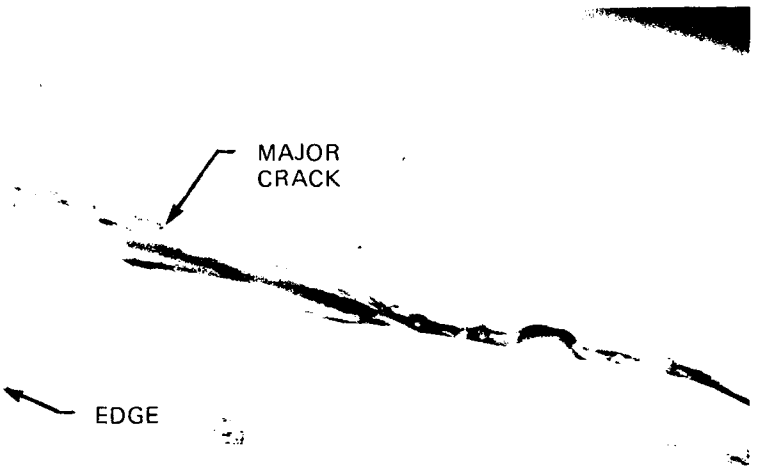


Figure 8. Crack growth in PC upon contact with DS2.

further chain cleavage and crack formation. While the critical strain in DETA is close to that in DS2, PC failed by developing a large number of stable crazes upon contact with DETA, as shown in Figure 9. The resulting damage, therefore, was less severe. Under a similar strain level, MCEL gave the highest critical value and rendered the least amount of damage, characterized by the development of a single stable craze.

It has been demonstrated that the toughness of a glassy thermoplastic polymer increases with its molecular weight, and there is a critical molecular weight (M_c) below which a polymer exhibits a very low fracture stress and low fracture toughness.[8,9] The craze fibrils (and therefore crazes themselves) are not stable below M_c. Consequently, the time it takes for cracks to form in PC in the presence of DS2 could depend on how fast the molecular weight drops to the critical value. Since the external stress facilitates the chain cleavage by lowering the energy barrier

8. KRAMER, E.J. "*Molecular Theory of the Fracture Toughness of Low Molecular Weight Polymers*." J. Mater. Sci., v. 14, 1978, p. 1381.
9. DONALD, A.M. and KRAMER, E.J. "*Molecular Weight Distribution Effects on Craze Micromechanics*." Polymer, v. 24, 1983, p. 1063.

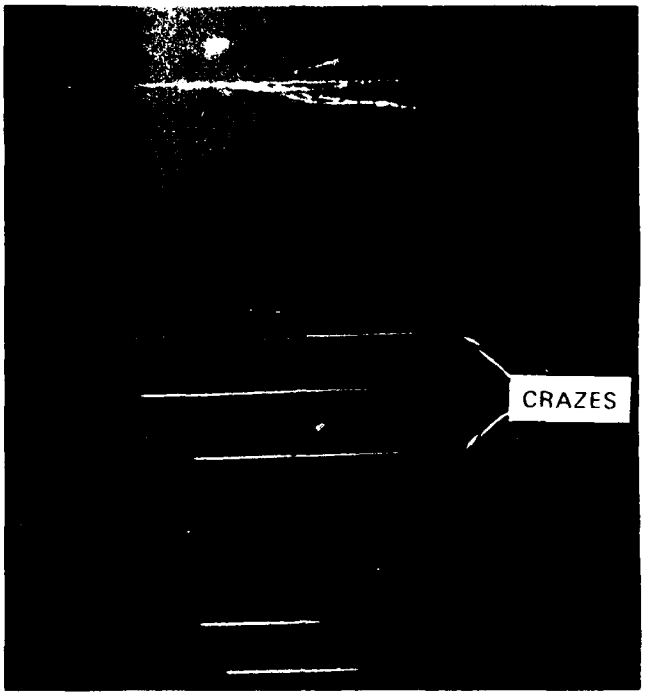


Figure 9. Craze growth in PC upon contact with DETA.

for the process, the failure initiation time is a function of the level of the applied stress and the rate of reaction between the carbonate groups and the attacking species. As a result, the prediction of the stress cracking behavior of PC in DS2 requires not only an understanding of the solubility of the products, but also knowledge of the kinetics of the reaction involved. A study of this behavior could be very complicated, and is beyond the scope of this work. The results, however, show that, above a strain level of about 0.32%, PC forms crazes and cracks in a few seconds or minutes after the application of DS2. When the strain was much higher than 0.32%, cracks formed instantaneously.

It is clear that the uncoated PC would not perform satisfactorily in the field in the event of chemical warfare due to its poor chemical resistance to the liquid simulants of CW agents[1] and DS2. Previous work has demonstrated, however, that the chemical resistance and stress cracking behavior can be improved by applying a coating material, Parylene C. The coating preserves the physical integrity and optical clarity of PC, and, furthermore, its intrinsic high toughness and good adhesion properties improve the stress cracking resistance of coated PC in DS2 significantly. At a strain level where the uncoated material failed almost immediately, stress cracking was prevented in Parylene C coated PC.

CONCLUSIONS

The results of this work have shown that the loss of physical integrity of PC in DS2 is due to the attacking of the PC carbonate group by the chemical components of DS2, resulting in chain cleavage. DETA appears to be the most aggressive component in DS2 causing the chain cleavage reactions. NaOH also plays an important role in the reaction; however, the overall eroding strength of DS2 does not seem to

be affected by the presence of NaOH. MCEL, while being rather inert to PC itself, seems to function as a carrier for NaOH. Mass spectroscopy indicates that a large number of small molecular weight products are formed as a result of the chemical interactions between DS2 and PC. The stress cracking resistance of PC is significantly lowered in the presence of DS2 and its components. It is suggested that prediction of the stress cracking behavior of PC in DS2 requires not only knowledge of the solubility parameter theory, but also that of the kinetics of the reaction involved. The chemical resistance and stress cracking behavior can be significantly improved by coating PC with Parylene C coating material.

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U.S. Army Materials Technology Laboratory
Watertown, Massachusetts 02172-0001.
CHEMICAL DEGRADATION AND STRESS CRACKING
OF POLYCARBONATE IN DS2
Lidia A. Lee and Janice J. Vanselow -

Technical Report MTL TR 87-46, July 1987, 13 pp -
illus-table, AMCMS Code 612105.H840011,
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Key Words

Polycarbonates
Stress cracking
Chemical degradation

Polycarbonate (PC) undergoes loss of physical integrity and ultimate dissolution in the decontaminant DS2. In order to gain a better understanding of this process, this study evaluates the effects of each of the three components of DS2 on PC. Weight loss measurements of PC in DETA (70%), MCEL (28%) and MCEL+NaOH (2%) show that DETA is the most aggressive component in DS2. The FTIR spectra of PC/DETA, PC/MCEL, and PC/MCEL+NaOH indicate that the principle degradation mechanism of PC in DS2 is the chain cleavage reaction taking place at the carbonate linkage due to the attack of the amine and the hydroxide. Mass spectroscopy analysis on the dry PC/DS2 residue indicates that numerous low molecular weight products are formed. Results of stress cracking of PC with DS2 and DETA show a considerable breakdown in resistance. The MCEL component is less aggressive than DETA, but still induces significant stress cracking.

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